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**HIGH-PRESSURE HYDROGEN TESTING OF SINGLE
CRYSTAL SUPERALLOYS FOR ADVANCED ROCKET
ENGINE TURBOPUMP TURBINE BLADES**

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Materials and Processes Laboratory

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16. ABSTRACT A screening program determined the effects of high-pressure hydrogen on selected candidate materials for advanced single crystal turbine blade applications. The alloys chosen for the investigation were CM SX-2, CM SX-4C, Rene N-4, and PWA1480. Testing was carried out in hydrogen and helium at 34 MPa and room temperature, with both notched and unnotched single crystal specimens. Results show a significant variation in susceptibility to Hydrogen Environment Embrittlement (HEE) among the four alloys and a marked difference in fracture topography between hydrogen and helium environment specimens.					
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TECHNICAL MEMORANDUM

HIGH-PRESSURE HYDROGEN TESTING OF SINGLE CRYSTAL SUPERALLOYS FOR ADVANCED ROCKET ENGINE TURBOPUMP TURBINE BLADES

INTRODUCTION

The selection of the present SSME turbine blade alloy, MAR-M246(Hf)DS, was based to a great extent on its performance in material development programs for aircraft gas turbines. However, the operating environment of a rocket engine turbine requires material properties which differ in certain respects from those required for an aircraft gas turbine. Candidate materials for the next-generation SSME turbine blades are being evaluated by criteria more specific to their unique application.

One significant difference between rocket engine turbines and aircraft gas turbines is the use of hydrogen fuel rather than petroleum distillates. This creates an aggressive rocket engine operating environment in which high strength materials may exhibit hydrogen environment embrittlement (HEE). Until recently, however, little attention was given to the effect of a hydrogen environment on the mechanical properties of turbine blade materials. As part of the effort to select candidate materials best suited for the rocket engine, Marshall Space Flight Center initiated a contract with Oak Ridge National Laboratory to establish facilities for high pressure hydrogen testing.

OBJECTIVE

This program was designed strictly for screening purposes. Its goal is to rank the performance of candidate single crystal turbine blade materials under hydrogen conditions. Although researchers are attempting to understand the mechanism and the factors that influence hydrogen embrittlement of various alloys, as yet there is insufficient information to predict HEE susceptibility. Consequently, a hydrogen screening program such as this is indispensable.

Ultimately, all candidate materials are to be tested in this program. Those alloys which have demonstrated improved properties as a result of modified heat treatments will be retested, and MAR-M246(Hf), both DS and single crystal, will provide a baseline for comparison.

EXPERIMENTAL PROCEDURE

The four materials tested initially, PWA 1480, CM SX-2, CM SX-4C and Rene N-4, are all single crystal superalloys which have demonstrated good mechanical performance in air. All were received in a heat-treated condition. The CM SX-2 and CM SX-4C material had received the standard heat treatment rather than the recently developed ONERA heat treatment [1]. The PWA 1480 material had been heat treated according to Pratt and Whitney's modified regime, and the Rene N-4 received its normal heat treatment.

The test apparatus (Fig. 1) is located in an underground pit, with only the servo-hydraulic control instrumentation (a standard MTS system) and the hydrogen supply on the ground floor above. The system may be isolated, evacuated and

purged with helium to flush out air and moisture. Then hydrogen or inert test gas is introduced and raised to 34 MPa within the autoclave by means of a booster pump. The specimen is mounted in the specimen grips; these, in turn, are mounted to the cap of the autoclave. A spherical bushing is used to maintain specimen alignment.

Both smooth and notched specimens of each alloy were tested (Fig. 2). The notched configuration was designed with a stress intensity factor of 8.0. All specimens were stress relieved at 850°C for 8 hr.

Three specimens of each alloy were tested in each of the categories in the test matrix (Table 1). Cross-head speed in all cases was $2.12 \mu\text{ms}^{-1}$, and the helium and hydrogen tests were conducted at 34 MPa and room temperature. The results which follow are the mean value of the three tests. In most cases, the coefficient of variation is less than 5 percent, indicating good consistency.

RESULTS

Smooth tensile testing shows only a small difference between the helium properties and those obtained in air for both ultimate and yield strength (Table 2). The difference between the UTS values and yield strength is an indication of the alloy's ductile behavior in helium. Hydrogen yield strength is not significantly different from that in helium; but the impact of the hydrogen environment is seen in the ultimate tensile strength, which is virtually identical to yield strength in hydrogen. This represents a severe loss of ductility. As soon as the specimens begin to yield, they fail. In the smooth tests, all alloys performed comparably, showing no obvious ranking of performance among the four alloys.

Reduction in area measurements on the smooth specimens reveal the extent of the ductility loss in HEE: approximately 50 percent in most cases. But it is in the notched strength that the difference among the alloys is clearly seen (Table 3). When the results are expressed as a ratio, the degradation of performance in hydrogen is most apparent for standard heat treated CM SX-2, and the alloys are easily ranked, with PWA 1480 showing the least loss of ductility.

An inspection of the fracture surfaces supports this ranking (Fig. 3). The high-pressure helium notched fracture surfaces show very little difference among the alloys. They are all predominantly ductile, reflecting the similar notched strengths. However, the hydrogen surfaces clearly differ. CM SX-2 shows an almost entirely brittle fracture, representing nearly instantaneous failure. As the notched ratio increases, the fracture surfaces show increasing areas of ductility.

A closer look at the fracture surfaces by the scanning electron microscope shows some ductility at both the edge and the center of the CM SX-2 specimen in helium (Fig. 4). In hydrogen, the surface is cleavage fracture. Note the orthogonal cracking in the last-to-fail region, correlating to a crystallographic fracture plane. In helium, the fracture surface of CM SX-4C is similar to that of CM SX-2 (Fig. 5). All the samples tested in helium show similar characteristics. In the hydrogen specimen both cleavage and ductility can be seen, corresponding to this alloy's better notch ratio. The properties of Rene N-4 are better still; cleavage is apparent at the edge, with a larger ductile area in the center (Fig. 6). Cleavage is less pronounced in PWA 1480, even at the edge (Fig. 7). Thus, there is a direct correspondence between the notched ratio and fractography of a specimen, and material performance in HEE can be clearly ranked.

CONCLUSIONS

This screening program is not conclusive in that it is not yet possible to specify one recommended alloy for use in SSME turbine blades. Work by other researchers has indicated that the ONERA heat treatment may significantly improve hydrogen properties [2]; therefore, it would be premature to rule out the alloys in this study which showed poor performance. However, several conclusions can be drawn at this point. First, susceptibility to HEE differs dramatically among superalloys. Furthermore, mechanical testing in atmospheres other than hydrogen yields no useful information for predicting material performance under actual engine operating conditions. Therefore, hydrogen testing is essential in an SSME turbine blade superalloy screening program.

REFERENCES

1. Kahn, T. and Caron, P.: Effect of Heat Treatments on the Creep Behavior of a Ni-Base Single Crystal Superalloy. Fourth RISO Int. Symp. on "Metallurgy and Mat. Science," Roskilde (Denmark), September 5-9, 1983.
2. Chandler, W. T.: Materials for Advanced Rocket Engine Turbopump Turbine Blades. Contract NAS 3-23536, Third Quarterly Technical Progress Narrative, July 28, 1983.

TABLE 1. TEST MATRIX

SPECIMEN ^a	ENVIRONMENT ^b	TEMPERATURE
NOTCHED	He	R. T.
NOTCHED	H ₂	R. T.
SMOOTH	He	R. T.
SMOOTH	H ₂	R. T.
SMOOTH	AIR	816°C

^a CROSSHEAD SPEED – $2.12 \mu\text{ms}^{-1}$; NOTCH – $K_T = 8$; STRESS RELIEVED AT 850°C FOR 8 HOURS – VACUUM

^b HELIUM AND HYDROGEN TESTS – 34 MPa; O₂ (H₂) << 10 ppm

TABLE 2. ULTIMATE TENSILE AND YIELD STRENGTH OF SMOOTH SPECIMENS

ALLOY ^a	UTS ^b (MPa)			Y. S. (MPa)		
	AIR 816°C	He 20°C	H ₂ ^c 20°C	AIR 816°C	He 20°C	H ₂ ^c 20°C
CM SX-2	980.9	1046.1	928.2	862.9	959.7	928.2
CM SX-4C	1004.2	1087.4	967.4	870.5	995.5	967.4
RENE N-4	969.0	1157.8	970.6	854.6	976.8	970.6
PWA 1480	995.4	1151.7	1047.2	916.5	1041.8	1047.2

^a ALL SPECIMENS STRESS RELIEVED AT 850°C FOR 8 HOURS/VACUUM

^b CROSS HEAD SPEED OF $2.12 \mu\text{ms}^{-1}$

^c OXYGEN CONTENT << 10 ppm

TABLE 3. REDUCTION IN AREA OF SMOOTH SPECIMENS AND RESULTS OF NOTCHED SPECIMEN TESTS

ALLOY ^a	R. A. ^b (%)		NOTCHED STRENGTH ^c (MPa)		NOTCHED RATIO
	He	H ₂	He	H ₂	H ₂ /He
CM SX-2	14.3	6.4	1498.5	215.9	0.14
CM SX-4C	10.6	8.6	1536.6	546.8	0.36
RENE N-4	10.6	5.1	1472.8	677.6	0.46
PWA 1480	12.4	6.5	1518.0	739.6	0.49

^a ALL SPECIMENS STRESS RELIEVED AT 850°C FOR 8 HOURS/VACUUM

^b FINAL CROSS SECTIONAL AREA ASSUMED ELLIPTICAL

^c NOTCH $K_T = 8$

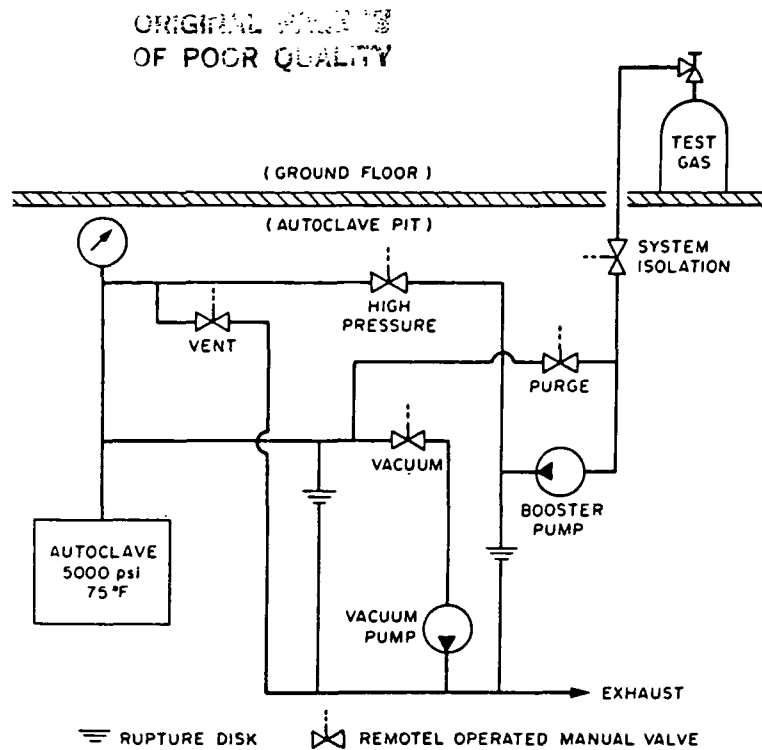


Figure 1. Flow schematic for high pressure mechanical properties test facility at Oak Ridge National Laboratory.

NOTES

1. ALL DIAMETERS CONCENTRIC WITHIN .005 FIR
2. ALL DIMENSIONS IN INCHES
- ③ SLIGHT TAPER TO CENTER PERMITTED
- ④ DO NOT UNDERCUT RADIUS

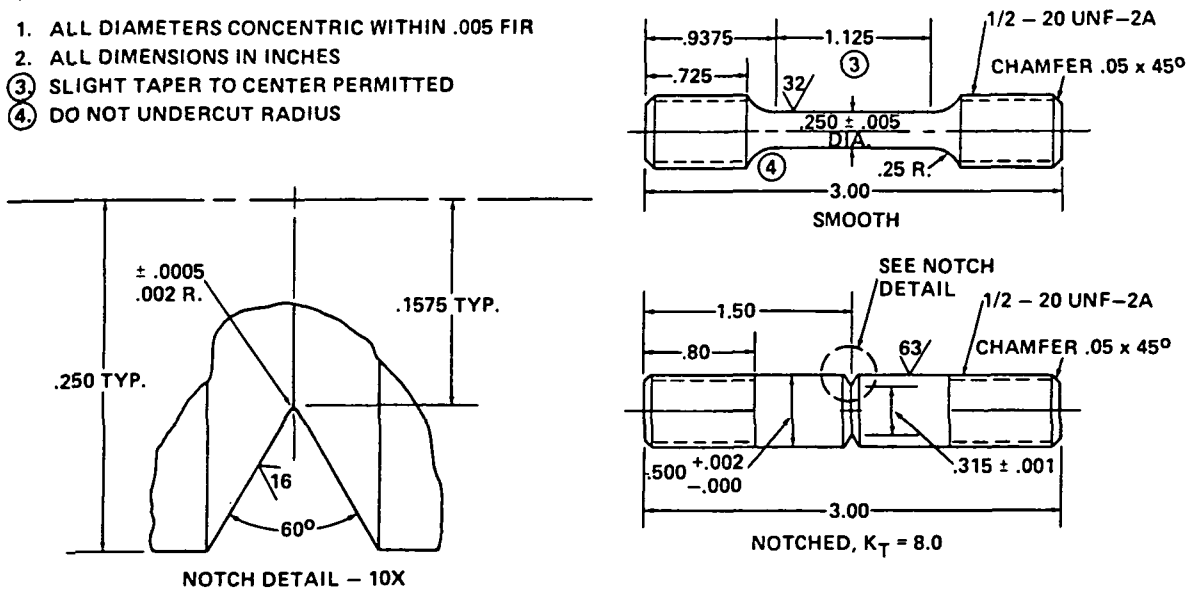


Figure 2. Smooth and notched tensile specimen configuration.

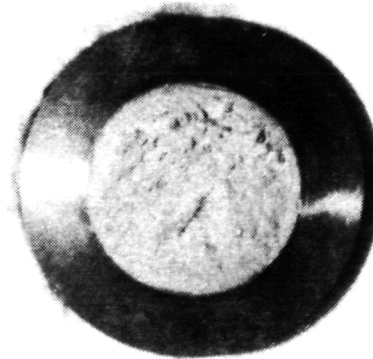
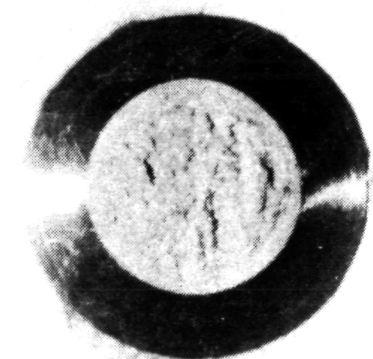
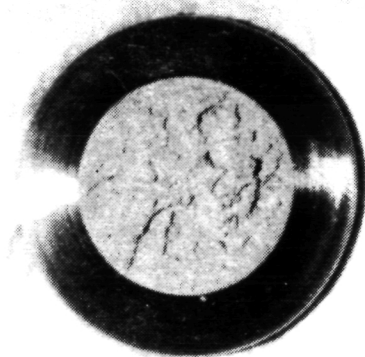
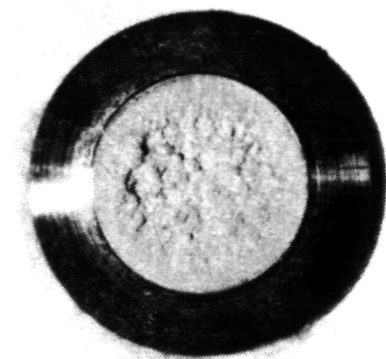
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CM SX-2

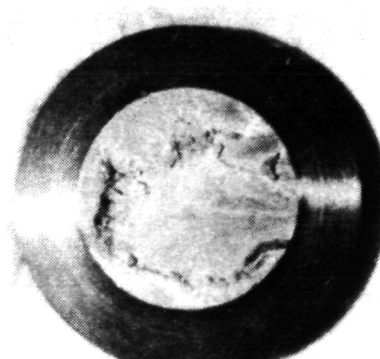
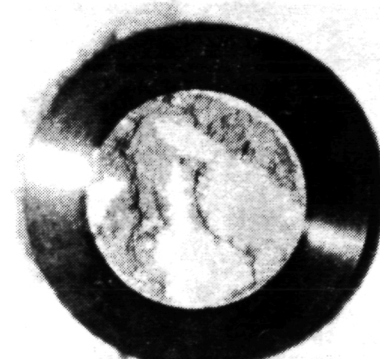
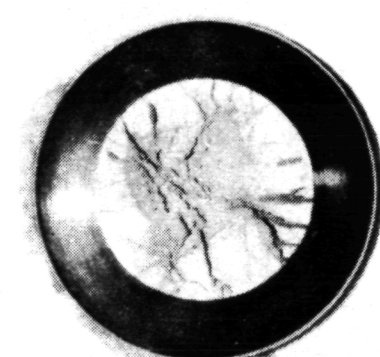
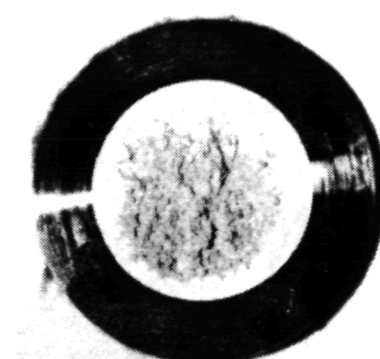
CM SX-4C

RENE N-4

PWA 1480



34 MPa HELIUM



34 MPa HYDROGEN

Figure 3. Notched tensile fracture surfaces. (4X)

CM SX-2

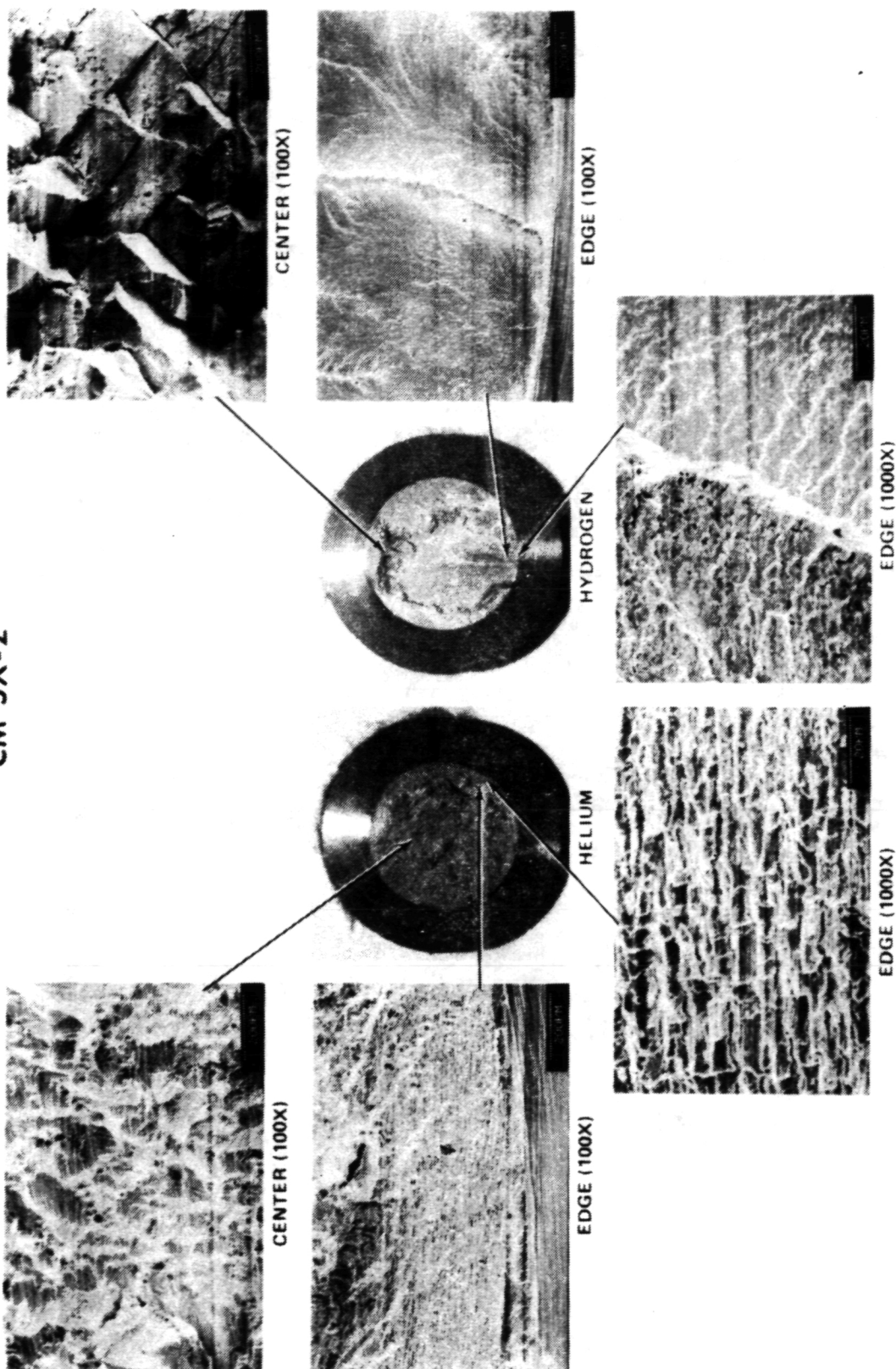
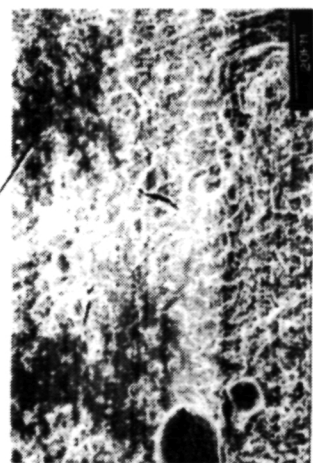
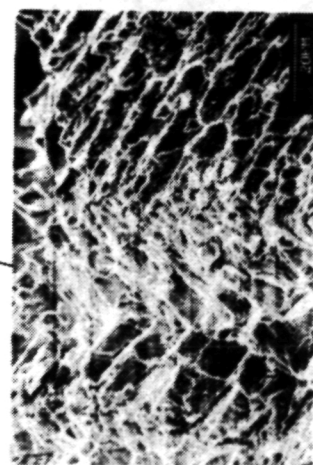
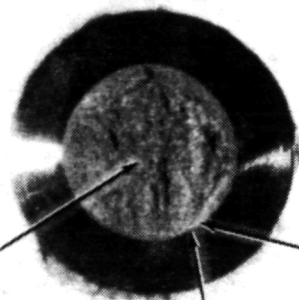
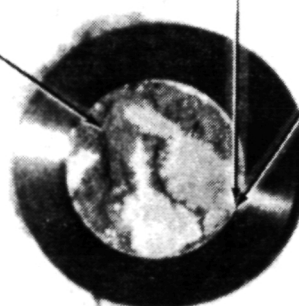


Figure 4. SEM fractography of CM SX-2 notched specimen.

CM SX-4C



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Figure 5. SEM fractography of CM SX-4C notched specimen.

RENE N-4

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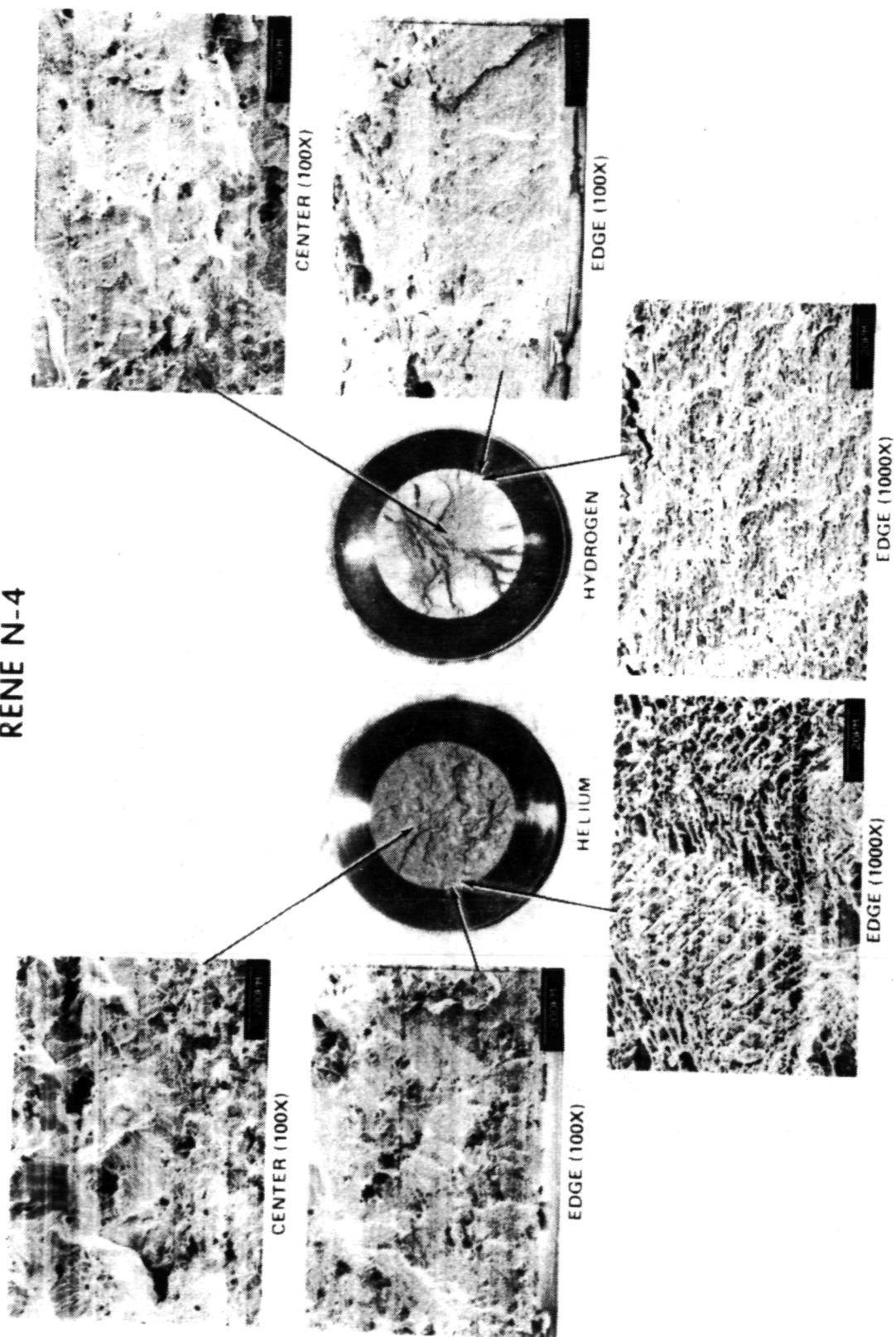


Figure 6. SEM fractography of Rene N-4 notched specimen.

PWA 1480

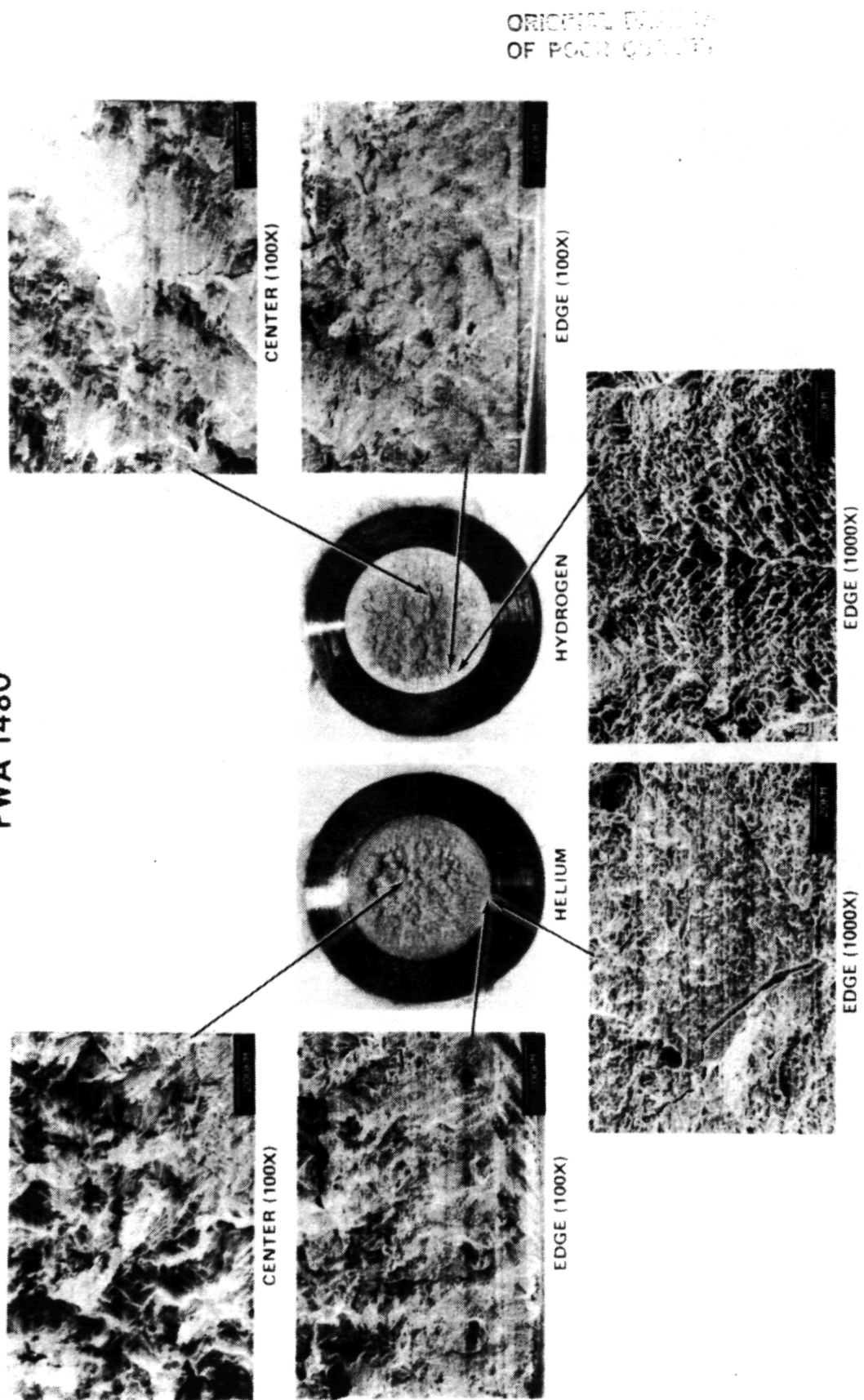


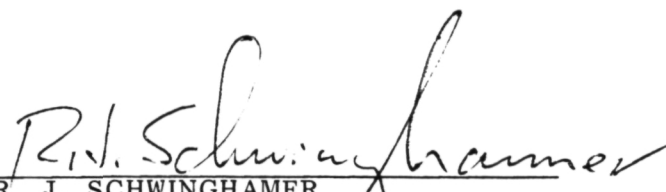
Figure 7. SEM fractography of PWA 1480 notched specimen.

APPROVAL

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The information in this report has been reviewed for technical content. Review of any information concerning Department of Defense or nuclear energy activities or programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.


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Laboratory